

HYDRO-PROCESSING OF HYBRIDIZED USED COOKING OIL INTO HYDROGENATION DERIVED RENEWABLE DIESEL USING LOCAL HETEROGENEOUS CATALYSTS

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ABSTRACT

The search for alternate energy to proffer permanent solutions to energy crises, fossil fuel depletion, and global warming is a pressing task. In this study, hybridized used cooking oil (UCO) was explored for hydro-processing using locally sourced biowaste catalyst. The coal fly ash (CFA) heterogeneous catalyst was reinforced into silica oxide (SiO₂) in a ratio of 60 wt% to 40wt%. The Parr reactor was used for hydroprocessing and conversion of UCO into hydrogenation-derived renewable diesel (HDRD). The value of the micropore volume of -0.0001 cm³ increased to 0.0014 cm³, and the external surface area increased from 0.8611 to 41.2571 m²/g, total surface area 0.5928 to 45.2771 m²/g and pore volume of 0.0053 increased to 0.1564 cm³/g. This property showed the potential biowaste catalyst for hydrogenation. The fractionated bio-crude product known as HDRD exhibits excellent fuel properties than conventional biodiesel. The total yield of bio-crude product was 67.15%. The product's high yield and excellent quality confirmed the potentials of CFA reinforced with SiO₂ as a suitable catalyst. Hydrogenation of UCO into HDRD using an optimal catalyst is a promising technology that will enhance commercialization addition, and the product is carbon-free, environmentally friendly, and economically viable.

KEYWORDS: CFA, Hybridized UCO, Hydro-Processing, HDRD & Fuel Properties

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1. INTRODUCTION

Increasing concerns for environmental pollution, global warming, persistent depletion of fossil fuel reservoirs, and greenhouse gas emissions have reinvigorated the search for alternative renewable energy sources. The ever-increasing applications of petrol-based fuel in compression ignition (CI) engines for transportation, domestic, commercial, and industrial purposes, and the increasing population, modernization, industrialization have become a challenge in meeting their energy needs [1]. The compression ignition (CI) engine exhaust emissions of fossil fuel account for a significant percentage of harmful environmental pollution and consequently increase global warming [2]. These challenges have engaged governments and scholars to search for a suitable alternative to mitigate petroleum products' adverse effects on the environment.

Green diesel otherwise known as hydrogenation-derived renewable diesel (HDRD), is a biofuel that possesses a superior property to biodiesel and fossil diesel. The conversion of vegetable oil to HDRD by hydro-processing technology removes oxygen from feedstock and replace it with hydrogen to produce a fuel similar to petroleum-based fuels. This can be achieved by placing a feedstock in a reactor in the range of 300 °C–400 °C and pressure in the 5–80 MPa with a heterogeneous catalysts in the presence of hydrogen gas for conversion of UCO into green diesel (mainly n-C₁₇H₃₆ and n-C₁₈H₃₈) [1, 3-5]. Biofuel can be processed using a desulphurization unit of an existing refinery with essential modification of the catalyst bed [6]. Organic liquid fraction (OLF) is obtained

when the bio-crude is distilled at different temperature ranges, producing a product of paraffin hydrocarbon with a high cetane number and high calorific value. The fuels also have sulfur less than 5 mg/kg, aromatic content of less than 1%, nitrogen less than 2 mg/kg, and olefin less than 0.2% double bond-containing components [7, 8].

The catalyst used in this study enhances the reaction mechanism rate for the conversion of triglycerides to a high yield of hydrocarbon products [9]. This can be achieved by removing heteroatom elements, breaking down the unsaturated molecules, and removing the acidity component. Micro-physical properties and ionic composition of a catalyst affect the activity for the production of HDRD. CFA reinforced with SiO_2 was used as a biowaste catalyst for the hydroprocessing of triglycerides[10].

Singh *et al.* [4] reported that CaO wt% is the highest elemental composition of BBTPPFS catalyst. Therefore, adding more CaO (wt%) in BBTPPFS catalysts could increase biofuel yield and its properties[4]. Silica oxide (SiO_2) is next to CaO in the elemental composition of BBTPPFS in weight percentage of 27.87%. Silica oxide can be found in red mud, fly ash and eggshell, etc. Silica oxide (SiO_2), also known as silica, is a natural compound made of two of the earth's most abundant materials: silicon (Si) and oxygen (O_2). Silica oxide is often known in the form of quartz. The earth's crust is 59 percent silica. Therefore, this novel study investigates the viability of hydro-processing a hybridized UCO by mixing silica oxide and freshly prepared CFA catalysts appropriately to explore the effect on the yield and characteristics of biofuel and their corresponding organic liquid fractions products. This was analyzed as per America Society of Testing and Materials (ASTM) standards. Locally sourced catalyst and UCO resources can be adopted for the production of HDRD which is the motivation of this study. This work aims to produce biofuel at the range of C_{15} - C_{18} using hybridized Used cooking oil and locally sourced catalyst generated from biowaste. At the same time, the scope is limited to reinforcing SiO_2 into BBTPPFS to obtain a higher yield of HDRD products. There is an increase in the global consumption rate of vegetable oil in recent times, as shown in Figure. 1. This is evidence of the availability and sustainability of UCO worldwide. [17]

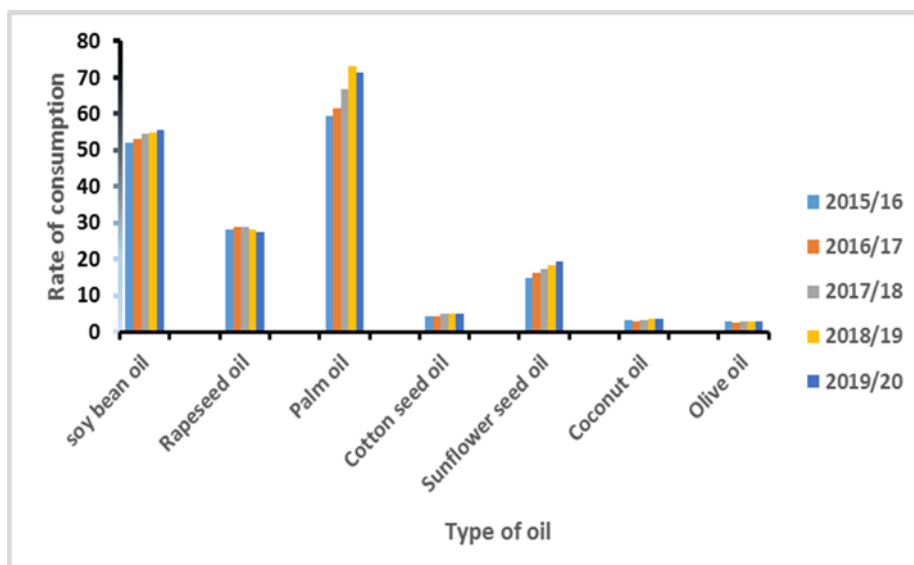


Figure 1: The Global Consumption by oil type 2015/16 to 2019/2020.

1.1. Used Cooking oils as Potential Feedstock

The global consumption rate of vegetable oil shows a tremendous increase in population, as shown in figure.1 [11]. However, Figure.1 is evidence of cost-benefit, availability, and sustainability of used cooking oil across the globe.

Table 1: Annual Consumption of Vegetable oil Per Capita

Country	Population/ 2020	Consumption(kt)	Per capita Food
United State	331,002,651	14684	39.2
North America	368,869,647	48824	25.8
Canada	37,742,154	1108	26.5
Europe	447,710,000	27617	23.2
Australia	25,694,393	641	26.6
Japan	126,027,215	2236	17.6
South Africa	59,308,690	1246	22.0
Malaysia	32,365,999	4335	24.8
Turkey	85,384,918	2283	30.0
Russia	146,007,035	3387	23.7
Mexico	130,501,308	2382	19.5
Brazil	214,310,027	6718	21.1
Iran	83,992,949	1739	19.6
Indian	1,380,004,385	18885	14.8
Pakistan	220,892,340	3762	19.8
Saudi Arabian	34,813,871	512	17.6
Indonesia	273,523,651	10814	19.6
Argentina	45,195,774	3384	29.7
Algeria	43,851,044	62015.7	21.8
Korea	51,269,185	1073	21.8
Egypt	102,334,404	1832	22.1
Sources: USDA February 2013 for vegetable oils and Wikipedia for population			

Canada consumes about 1108 metric kiloton of UCO per annum [12, 13]. About 600 000 tons of UCO have been collected annually from bakeries, takeaway outlets, and restaurants in South African [14, 15]. The UCO for this research is sourced from fast-food outlets used for frying chicken and chips in South Africa.

The available source and sustainable feedstock for the production of HDRD are UCO [16]. The huge generation of UCO across the globe as reflected in table 1 shows the sustainability of UCO resources when harness for the production and commercialization of biofuel.

1.2 Biowaste Catalyst

Various scholars have studied several chemical agents to search for the catalyst that has the potential to produce biodiesel and HDRD. They found out that the conversion of triglycerides depends on selecting a suitable catalyst with adequate properties to support the production. Choice of catalyst plays a vital role in the production processes and the hydrocarbon product during hydrogenation. It will also depend on the reaction conditions, i.e., temperature, pressure, liquid space hour velocity (LSHV), and H₂ to oil (volume/volume) ratio [17].

Marker et al. [18] researched various catalysts that can drain out the water and produce hydrogen. Applications of commercial catalysts have contributed to significant setbacks witness in the commercialization of green diesel. Munoz et al.[19], Abdullah et al.[20], and Yao et al.[21] reported the utilization of a catalyst derived from biomass-based waste for hydrocarbon production, while Stanković et al.[22], Yusup et al. [23] have suggested various methods, technologies, benefits, and shortcomings of bio-based fly ash catalysts for biofuel production. Apart from the abundant and sustainable CFA resources in South Africa, the choice of biowaste catalyst for this research is to make the value chain green in the process of production and commercialization of biofuel. This research aims to investigate hydro-processed hybridized used

cooking oil into Hydrogenation Derived Renewable Diesel (HDRD) using two reinforced heterogeneous catalysts. This research also examines the effect of the addition of SiO_2 to CFA catalysts in varying proportions on the hydro-processing of hybridized used cooking oil and explore the yield of biofuel and its properties. However, the motivation for this study is the use of biowaste and hybridized used cooking oil to produce green diesel. The product is characterized as per ASTM/EN standard and compares with FAME, petrol-based diesel.

2.0 MATERIALS AND METHOD

The UCO samples were collected from three takeaway outlets in Durban, South Africa, just before disposal. The profile and chemical composition of UCO was analyzed, as shown in Table 1. All the chemicals used are of analytical reagent grade. The catalyst (silica oxide, SiO_2) was procured from Lichro Chemical and Laboratory Supplier, Durban, South Africa, and CFA sourced from ESKOM SOC Limited–Lethado Power Station and calcinated in the muffle furnace at 550°C for two hours. The chemical analysis of both the catalysts was done by the Brunauer-Emmett-Teller (BET) and Spectroscopic analysis (FTIR).

2.1 Brunauer-Emmett-Teller (BET)

Three samples consisting of 0.25g were subjected to Micromeritics ASAP 2460 devices for analysis. Water is removed from the samples at the temperature of 100°C under nitrogen gas for twenty-four hours. Then, the samples were allowed to cool gradually in a vacuum to make their surfaces and pores area open for close analysis. The surface area of the sample (m^2/g) was determined by the BET method, while the pore size, total pore volume cm^3/g at Standard temperature pressure and average pore radius (\AA), were estimated using the Barrett-Joyner-Halenda (BJH) method. BET analyses outcome showed that the pore volume, surface area, and pore area, possess the potential green catalyst to mild crack UCO into green diesel.

2.2 Spectroscopic Analysis

The devices (Perkin Elmer Co., USA, model 15.011) is used for the analysis of seven samples to obtain a good absorption spectrum, the dilution and homogenization of the seven samples with spectroscopic grade. The discs with 12.7 mm ID and ≈ 1 mm thick, 10 tons manual hydraulic press less than 60 s.

The measurement of the spectrum between 300 to 4000 cm^{-1} was done and recorded on a spectrometer of model system 1000 FTIR with a resolution of 2.0 cm^{-1} . The utilization of CFA as a potential's catalyst for conversion of UCO into HDRD depended on the location and peaks from the sample spectra.

2.3. Production of Biofuel

The production of HDRD from UCO was carried out in a Parr reactor having a mechanical stirrer, pressure and temperature sensor heating unit, reading gauge. For this research work, a biowaste catalyst was used after pulverizing and calcinated in a muffle furnace at 550°C . The novelty of this work is the in-situ hybridization of the feedstock, which comprises WSF_C , WPOF_C , and WPOS_C . Hybridization of feedstocks provides a straightforward, easy, cost-effective, and innovative way of adjusting and enhancing the physicochemical properties, thermal and spectroscopic behaviour of feedstock to enhance the suitability for biofuel production. The UCO sourced from the restaurant were blended in different proportions and analysed for properties determination the results shown a good property for hydrogenation. The hybridization of waste cooking oil was described in the authors' previous work [24]. The hydrocarbon production was

performed by introducing 300 ml of oil at 380°C, with 5 MPa initial hydrogen pressure and 90min reaction time using the catalysts. The reaction parameters were kept constant for every reaction. The reactor was allowed to cool at room temperature after the completion of the hydro-processing. Then, the total product is collected. The ratio of the mass of bio-crude obtained after separating the catalyst particle to the volume of feedstock introduced to the reactor is estimated.

The conversion of the products can be calculated by applying equation 1;

$$\text{Conversion (\%)} = \frac{\text{Feed(temp.)} - \text{Product(temp.)}}{\text{Feed(temp.)}} * 100 \quad [25] \quad (1)$$

Where: Feed(temp) and Product(temp) are the weight percent of the feed and product, respectively, which depend on the boiling point of the temperature of Diesel.

Applying equation 2 to determine the diesel selectivity.

$$\text{Diesel selectivity (\%)} = \frac{\text{Product}(\Delta\text{temp.}) - \text{Feed}(\Delta\text{temp.})}{\text{Feed(temp.)} - \text{Product(temp.)}} * 100 \quad [25] \quad (2)$$

Table 2: Catalyst Effect on Biofuel Yield								
Parameter Catalysts	The Volume of used Cooking Oil (%)	Temperature (°C)	H ₂ pressure (MPa)	Catalyst (g)	Catalyst (wt%)	Time (Min.)	Water Loss (%vol.)	Total Yield (%vol.)
SiO ₂	300	380	5	40	3	90	8.15	67.85
CFA	300	380	5	40	3	90	7.20	67.20
SiO ₂ +CFA	300	380	5	40	3	90	6.85	69.15

Table 2 shows the effect of catalysts on the oil yield and the parameter for hydro processing of used cooking oil; from the table, the highest product of 69.15% volume was obtained from SiO₂+CFA_E catalyst, while 67.85% and 67.20 % yield were obtained from SiO₂, and CFA_E respectively. The char of 9 %volume and 8 % volume of the feedstock volume charged into the reactor was obtained.

3. RESULT AND DISCUSSION

The hydroprocessing of hybridized UCO using heterogenous catalyst (CFA reinforced with silica oxide), under the reaction condition provided in figure 3 was done. The author's previous work reported the preparation protocol and the in-situ hybridization of used cooking oil of the novel feedstock [26]. The optimal catalyst, feedstock, and operating parameters were employed in a Parr reactor to produce HDRD. The chemical transformation that occurs during hydro processing in a reactor is hydrocracking, hydrodeoxygenation, isomerization, etc. The bio-crude is separated and fractionated to obtain green diesel while the residual fuel is collected at the base of the fractional distillation column. The water and gas formed during the hydrocracking process is evidence that oxygen is removed from the feedstock in form of H₂O, CO₂ and CO.

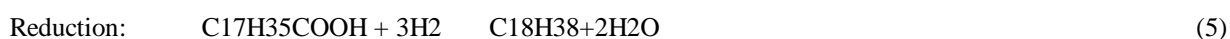
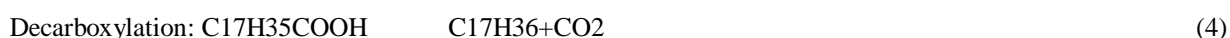
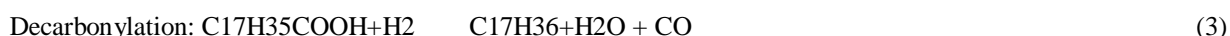


Table 1 shows the physicochemical properties of UCO. The fatty acid composition makes them viable as a potential feedstock for the production of green diesel. The available source and sustainable feedstock for the production of HDRD are UCO [18]. This feedstock was adopted for this research work.

Table 3: The Physicochemical Properties of Used Cooking Oil

Properties		Experimental Value	Standard Value	Method used	Reference
Acid Value (Mg of KOH/gm)		2.48	3.0 _{max}	AOCS Cd3d-63	[27]
Density @ 20°C (Kg/m ³)		919.7	923	ASTM D4052	[28]
Iodine value(cg/g)		51.5	100 _{max}	AOCS Cd 1-25	[27]
pH value		6.52	7.00	AOCS	[28]
Kinemematics viscosity@40°C		36.14	43.52	ASTM D446-12	[28]
Saponification value KOH/g		196.2	195 _{max}	AOCS Cd3d-63	[27]
Heating value MJ/kg		41.12		IS:1359-1959	
Fatty acid composition of	UCO				
Palmitic acid	C ₁₆ H ₃₂ O ₂	0.36			
Oleic acid	C ₁₈ H ₃₄ O ₂	0.8			
Stearic acid	C ₁₈ H ₃₆ O ₂	—			
Caprylic acid	C ₈ H ₁₆ O ₂	—			
Linoleic acid	C ₁₈ H ₃₂ O ₂	0.10			
Myristic acid	C ₁₄ H ₂₈ O ₂	—			
Linoleic acid	C ₁₈ H ₃₀ O ₂	4.5			
Elemental composition	(wt%)				
Carbon		75			
Hydrogen		12.4			
Nitrogen		0.20			
Oxygen		10.90			

3.1 BET Analysis

The BET analysis on the surface area, pore volume, pore size, external surface area, average pore radius, micropore volume, and micropore area are presented in table 4. The catalyst such as surface area, pore volume external surface area, and pore volume of CFA were significantly increased when SiO₂ with the values of 0.5928 m²/g, 0.8611 m²/g, and 0.00532 cm³/g is added to CFA with the value 35.1102 m²/g, 32.5753 m²/g, and 0.1281 cm³/g respectively. The addition of SiO₂ caused a reduction in the value of the average pore radius in the mesoporous range as a result of impurities. Hence, there was an increase in the micropore volume of SiO₂ from -0.000121 cm³/g to 0.000582 cm³/g. Vargas et al. [29] reported a value of 9.028 m²/g BET surface area and pore volume of 0.01055 cm³/g for biomass fly ash.

The incremental changes that occur in pore volume caused the formation of new mesopores, which can also be formed by the addition of a support's catalyst [30]. The BET surface area is a principal physical property that controls the catalytic activity of a solid particle, higher surface area of a catalyst enhances better catalytic activity and more interactions with the reactants [31]. The high BET surface area of 35.1102 m²/g will support the addition of SiO₂ and CFA's catalytic activity for the adsorption and desorption of triglycerides, glycerine, and HDRD [32]. The results of the BET analysis of the catalyst samples has shown their acceptability as a low-cost and biowaste catalyst for hydrogenation of UCO into HDRD [33].

The addition of SiO₂ to CFA was used for the hydroprocessing of hybridized used cooking oil because of its excellent thermal, spectroscopic, composition, and surface area of the coal fly ash. In addition, the biowaste catalyst poses the hydrocracking potentials to convert the feedstock to HDRD range of C₁₅-C₁₈.

Table 4: Properties of Heterogenous Catalyst for Hydrogenation

Properties	Unit	SiO ₂	CFA	SiO ₂ +CFA
BET surface area	m ² /g	0.5928	317.2404	35.1102
External surface area	m ² /g	0.8611	279.1870	32.5753
Average pore radius	Å	179.68	70.72	72.98
Pore volume	cm ³ /g	0.005326	1.1218	0.1281
Micropore volume	cm ³ /g	-0.000121	0.014827	0.000852
Micropore area	m ² /g	*NR	38.0533	2.5349

3.2 FTIR Analysis

The FTIR spectra analysis of the biofuel obtained from the SiO₂+CFA catalyst is shown in Fig. 3. The FTIR result shows that the trend of the wavelength bands is associated with the chemical functions of biofuel such as hydrocarbon and organic fatty acid on the thermo-chemical conversion of hybridized UCO into HDRD.

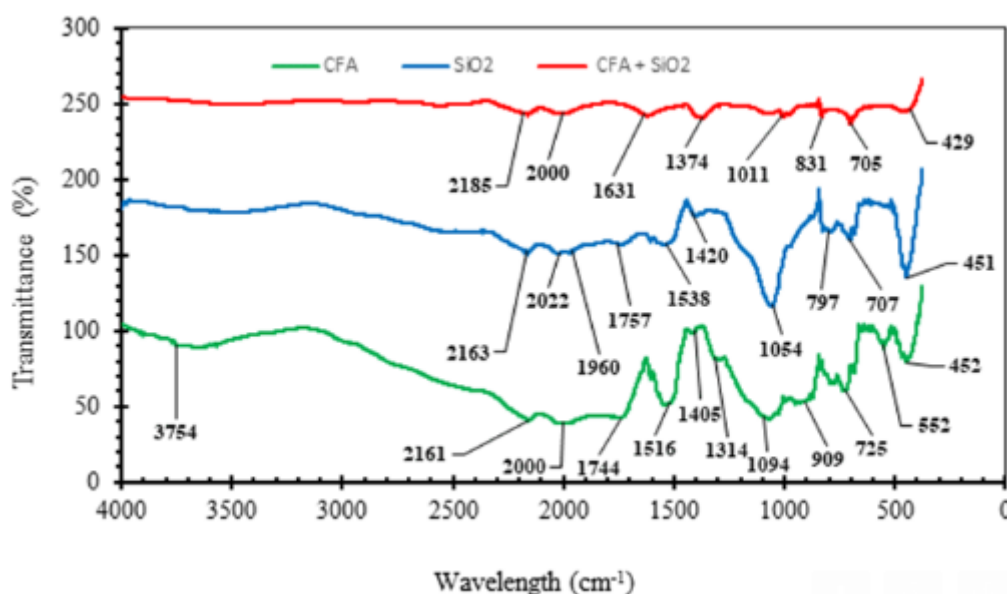


Figure 2: FTIR Spectra of Biofuel.

The wavelength peak at 1010 cm⁻¹ and 1420 cm⁻¹ have the potentials of the peculiar bands for tested hydrogenation catalysts [34]. FTIR spectroscopy confirms C₁₁-C₁₈ as the significant hydrocarbon, and other compounds like organic acids, aldehydes, and ketones are oxygenated compounds. Apart from this, the density (Carbon, Oxygen, Hydrogen, sulfur, and Nitrogen) contained in the biofuel reveals the high value of hydrogen and oxygen, which support UCO conversion into hydrocarbons [4].

The optimal catalyst, feedstock, and operating parameters were employed to produce HDRD in a reactor. The chemical transformation that occurs during hydroprocessing in a reactor is hydrocracking, hydrodeoxygenation, isomerization, etc. The bio-crude is separated and fractionated to obtain green diesel while the residual fuel is collected at the base of the fractional distillation column.

HDRD containing the highest percentage yield of 67.15 % volume, and volume the residue is 32.85 % obtained and separated from the remaining hydrocarbon. The optimal yield outcome obtained can be traced to the high percentage of silica oxide in CFA that enhanced the catalytic activities for the production of HDRD.

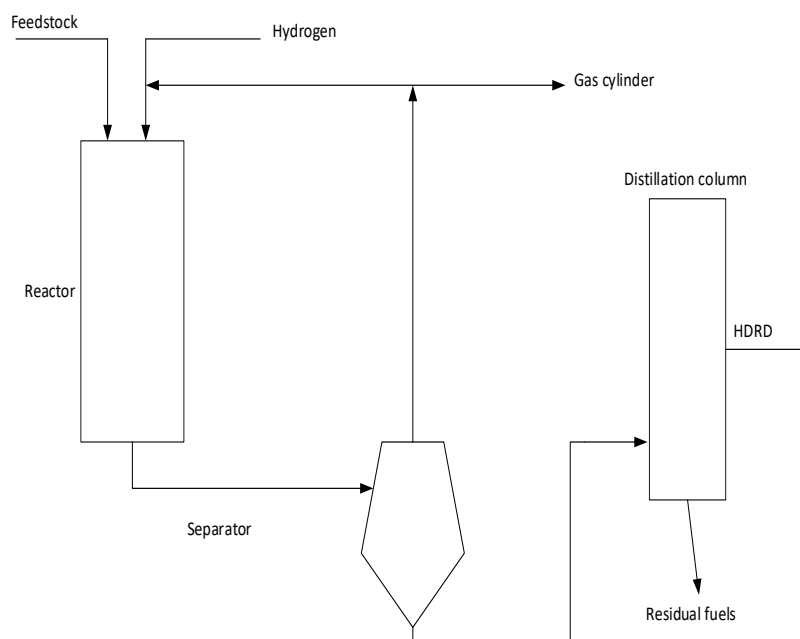


Figure 3: Flow Chart of Conversion of UCO to HDRD.

Table 5 Properties of Biofuel Produced by Catalyst

		SiO ₂ +CFA	SiO ₂	CFA
Properties	Unit	value	value	value
Density	g/mL	0.832	0.848	0.845
Kinematic viscosity	cSt	3.50-7	2.9	2.8
Cetane number	-	>90	85	87
Flash point	°C	85	81	83
Net heating value	MJ/kg	54	45	47
Water	% vol.	0	0	0
Calorific value	MJ/Kg	49.85	46.23	45.73

The HDRD produced has a superior property. Biofuel is free from impurities like sulfur, oxygen, nitrogen, and water compared with conventional petrol base fuel. The net heating value of petrol diesel is 42-43 MJ/kg while HDRD is 54MJ/Kg. The cetane number of the fuel is greater than 90, while petroleum diesel has less than 60. In addition, the oxidation stability of HDRD is very high and has a nominal acid value. The flashpoint of HDRD is significantly higher; also, a paraffinic compound of HDRD withstands long-term storage to biodiesel. Other contaminations like sulfur, nitrogen and aromatic contents are very low as compared to petrol-diesel. The cold flow properties of HDRD are better when compared with petrol-based diesel [4].

4. CONCLUSIONS

HDRD obtained by hydro-processed hybridized UCO using biowaste as a catalyst is feasible. In this research, the novel feedstock and the mixture of SiO₂ with CFA used to produce HDRD resulted in a high yield of 69.15 % volume with

excellent properties. Apart from the benefit of low cost, eco-friendly, availability, and sustainability of the production resources, it is highly effective for hydrogenation of UCO. The FTIR analysis of the biofuel reveals that the main product found in the optimal yield of HDRD are paraffinic hydrocarbons. Furthermore, HDRD has excellent properties (high cetane number, oxidation stability, kinematic viscosity, zero water, and zero acid value) compared to biodiesel and petrol-based diesel. The value chain of locally sourced catalyst and UCO, which form significant resources for the production of HDRD will enhance the commercialization of HDRD.

Conflict of Interest: No conflict of interest.

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